PREPARATION & CHARACTERIZATION OF POROUS CERAMIC BODY USING BALL CLAY

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Abstract - *In the past few years, porous ceramic substances having progressed microstructure has come to be very famous because of its extensive utility in exclusive fields. Now a days there are numerous strategies which include Polymeric Sponge Replica Technique, Starch consolidation technique, Gel Casting method and so on. Had been evolved for the training of porous ceramic frame with managed microstructure which includes porosity, pore length, pore connectivity and many others. In this venture paintings, an effort has been made to put together porous ceramic frame (the use of Ball clay as raw material) by means of Polymeric Sponge Replica Technique. Ball clay used as main raw material with a few amounts of dispersant (sodium silicate) and some samples additionally prepared with a few amounts of PVA as binder and also found the effect of binder on porosity. This microporous body prepared via sponge duplicate approach may be utilized in distinct regions which include molten metallic filtration, warm gases filtration and so on. Solid loading is the primary aspect of the slurry was determined for the education of porous ceramic frame of different pore morphology by using sponge replica technique. The microstructure which includes pore length, pore connectivity has been analysed by way of the FESEM.*

Key Words: Porosity, Sponge replica, viscosity, microstructure.

1. INTRODUCTION

Overview of Porous Ceramics Porous ceramics has been very famous in the latest years, due to its progressed microstructure inclusive of porosity and pore length distribution, good enough strength, low thermal conductivity and excessive permeability and so on. These porous ceramics have a whole lot of programs in technical discipline namely filtration of metallic and warm gases; ion exchangers, burner and many others (Al-Naib, 2018). Porous ceramics additionally observed extensive utility in scientific, mining, oil & gasoline exploration, chemical processing, pharmaceutical industries and so forth. Application location of porous ceramic depends at the composition and morphology of the porous structure, particularly pore length, shape and its distribution together with the pore connectivity. Porous ceramics substances are substantially used as filters, heat insulators, absorbers, catalyst supports etc. In specific fields of engineering. Application of porous ceramics strongly depends on the pore length, permeability, and precise floor place (S. Somiya). At present, the maximum notably used are the subsequent production methods of porous permeable ceramics: processing of mono-fractional beginning materials, foaming technique, approach of burning-out of additives, and chemical approach of pore formation. Each production technique of porous permeable ceramics has each benefit and essential risks as compared to different methods. Specifically, ceramics prepared through the foaming approach or by using the chemical technique of pore formation are characterized by way of low permeability because of predominantly closed porosity. Ceramics acquired by means of the approach of burning-out of components have inhomogeneous porous shape, due to the fact that getting a homogeneous distribution of additives within the volume of the mixture is hard. For this purpose, the look for new technologies of manufacturing porous ceramics is being continuously pursued. Clays are used as liner substances in drug shipping device and agrochemical delivery agents, and as catalytic substances (M.J.CarrizosaM.J.CalderónM.C.HermosínJ.Cornejo, 20 March 2000). They additionally play a vital position in biogeochemical strategies with the aid of retaining or releasing metallic vitamins within the soil. Clays encompass negatively charged aluminosilicate layers kept together through cations. The most characteristic belongings are their capacity to take up water among the layers, ensuing in robust repulsive forces and growth of clay (Maja Kokunešoskia, June 2014) Nowadays, membrane technology has been extensively hired in water and wastewater treatment procedure because of water shortage, excessive water prices and stricter regulations that required more superior water remedy era. Most of the available membrane inside the marketplace is polymeric primarily based membrane, and it's been extensively used in membrane method industry. Polymeric membrane has a decrease capital price, scalability and properly separation characteristics. However, they are low fouling resistance, low lifespan, low-temperature degrees and coffee corrosion resistance (Rathila, 2020). Opposite to ceramic membrane, can be applied in the acute environments because of their most important benefits, in super high chemical stability, thermal and mechanical resistance. The ceramic membrane also well-known for having a longer lifespan, ease of cleansing, low dielectric steady and a low thermal conductivity (A.J. Burggraaf, 1991). Ceramic membrane including metal oxides which includes alumina, titania, zirconia and others are most usually carried out, in particular manufactured from alumina. Alumina ceramic membrane is well-known in its terrific thermal, chemical and structural balance. However, it indicates a downside of high sintering temperature (a big quantity of warmth is needed) that's above to 1500 °C to achieve correct settlement among mechanical power and porosity. Besides, alumina itself is regarded as an excessive fee material, for this reason extremely luxurious ceramic membrane production. On the opposite hand, the funding fee of ceramic membrane an awful lot expenses as compared to the polymeric membrane. Thus, the fabrication of low-cost ceramic membrane based totally at the herbal clay (e.g., Kaolino-illitic clay, smectetic clay, Moroccan pozzolan clay) become studied by numerous researchers because of their abundance in nature. New flat ceramic microfiltration membranes were currently evolved from ample, herbal substances which includes herbal Moroccan bentonite for business wastewater remedy. In well known, clays from bentonite materials were implemented in diverse industrial fields including acts as catalysts, adsorbents and ion exchangers because of its chemical and physical houses. Bentonite substances have broad particular floor region, organic and inorganic ion adsorptive affinity, and cation change capability as properly (Fazureen Azaman a, 2021)

Open pore and ii) Closed pore.

The porous ceramic frame having open pores are used for filters and catalysts while the closed pores are required for thermal insulation. Pores additionally labelled into the following categories based totally on their sizes:

1) Microporous (50 nm).

2) Mesoporous (2-50 nm)

3) Macroporous (>50 nm).

Meso and microporous can be applied in molecular sieves and in catalysis [2]. Macroporous can be utilized in whitewares like roof tiles and also in advanced ceramics together with medicines and car engines (M.V.Twigg1J.T.Richardson2, 2002)

The development of the porous filters fulfilled the requirements like the healing of the methane from mines, expulsion of carbon dioxide and hydrogen sulfide from natural fuel, healing of hydrogen in petroleum refinery operation. In the foundry enterprise, porous filters are applied for molten metallic filtration [4]. Porous ceramics can also be utilized in sensors, battery substances as well as inside the field of biomedical. Types of porous ceramics primarily based on pore structure and their uses are given below table 1. (KHAN, 2015)

Table.1. Types of porous ceramics based on pore structure and their uses (Anne Julbe, 2001)

Pore Structure	Uses
Microporous and Mesoporous body	Coatings Sensors and Actuators Catalytic support Desiccant materials
Foam and Honeycomb pore structural body	Flue gas filters Molten metal filtration Burners Electrodes of fuel cells Porous scaffolds which are used in tissue engineering Kiln furniture.
Multilayer ceramic body	Ultra-filtration membranes Nano filtration membranes Hot gas filtration membranes Dense membranes Zeolite membranes

1.1 Fabrication Technique of Porous Ceramics

Different strategies had been advanced with a purpose to get porous ceramic frame of desired morphology and houses. The strategies are sacrificial template technique, paste extrusion technique, freeze casting method, direct foaming approach, sponge reproduction approach, fast prototyping method and so forth (Gang Liu, 2011).

1.2 Sponge Replica Technique

Pore size in the range of four hundred nm to four mm can be realized in a porous ceramics fabricated with the aid of sponge duplicate technique. In the sponge reproduction method, commercial polymeric sponge turned into used as template. The manner consists of coating of open-mobile polymeric sponge with stabilized slurry after which sintering of that coated sponge which yields an imitation of porous ceramic. This method produces ceramic body with a more a part of open mobile sponge microstructure. The pore morphology of ceramic frame fabricated by way of this method can be tailor-made with the aid of controlling the consistency of the slurry and the polymeric sponge characteristics (strut thickness, pores size, shape and its distribution). The strut thickness, and pore morphology depends on the thickness of ceramic slurry coating on sponge strut.

In the prevailing have a look at ball clay has been chosen because the matrix of porous ceramic. Precursor ball clay is characterised for its bodily and thermal behaviour. The amount of electrolyte requires to make a strong ball clay slurry became optimized. Slurry changed into organized with unique quantity of ball clay loading with taking optimized quantity of sodium silicate and the impact of solid loading on viscosity turned into studied. Effect of PVA addition at the houses of porous scaffolds has additionally been studied (KHAN, 2015).

2. EXPERIMENTAL PROCEDURE

Optimization of deflocculant for the formation of strong ball clay slurry Sodium silicate has been used as deflocculant on this present work. Slurry has been made with distinct amount of sodium silicate various from 0.1 to 0.9 wt.% with a fixed stable loading of 20 wt.%. The stability of slurry has been calculated through measuring the zeta ability, sedimentation top and viscosity of the slurries. Sedimentation peak was measured by means of pouring of slurry in a 100ml measuring cylinder. Slurry was filled with a top of 80ml and measured the sedimentation top at different c programming language of time as much as 24 hours of settling.

2.1 Optimization of binder for slurry of desired viscosity

PVA (polyvinyl alcohol) has been used as binder. Slurry has been made with a fixed solid loading of 20 wt.% together with a varying amount of PVA from 2 to 8 wt.% with the help of Rheometer, viscosity is measured.

2.2 Rheological Behaviour of Ball Clay with Deflocculant

Clay slurries were made up via utilizing ball clay and sodium silicate as a deflocculant with varying weight proportion. Slurries have been prepared with solid loading of 35 wt.%, 45 wt.%, 50 wt.% and 55 wt.% in conjunction with 0.4 wt.% sodium silicate as deflocculant. All these compositions of slurries are pulverized in a pot mill for 12 hours with a few alumina balls used as grinding media. Total weight of all compositions is 100gm.

Table.2. Batch Calculation

BATCH COMPOSITION				
Solid loading (wt.%)	**Wt. of water (gm)**	**Quantity of clay (gm)**	**Quantity of deflocculant (gm)**	
35	65	35	0.4 wt.%	0.14
45	55	45	0.4 wt.%	0.18
50	50	50	0.4 wt.%	0.20
55	45	55	0.4 wt.%	0.22

2.3 Slurry preparation with PVA

Clay slurries have been made up by using ball clay and optimized fee of sodium silicate as a deflocculant and with optimized wt.% of PVA as binder. The amount of PVA added turned into five wt.%, and with varying quantity Ball clay. Slurry become made with 35 wt.%, 45 wt.%, 50 wt.% and 55 wt.% strong loading of Ball clay along with optimized price of binder and deflocculant. All those compositions had been moist milled in pot mill for eight hours with some alumina balls used as grinding media.

Table. 3 Batch Calculation containing binder

BATCH COMPOSITION						
Solid Loading (wt.%)	**Quantity of water (gm)**	**Quantity of Ball clay (gm)**	**Amount of deflocculant (gm)**		**Amount of PVA (5w.t% PVA solution) (ml)**	
35	65	35	0.4 wt.%	0.14	5 wt.%	35
45	55	45	0.4 wt.%	0.18	5 wt.%	45
50	50	50	0.4 wt.%	0.20	5 wt.%	50
55	45	55	0.4 wt.%	0.22	wt.%	55

2.3.1 Different Techniques for the Preparation of Porous Ceramic Body

Porous Ceramic body can be made by three methods.

1) Starch consolidation method (SSM)
2) Sponge replica technique (SRT)
3) Combination of SSM and SRT

Porous ceramic body has been fabricated by sponge replica technique.

2.4 Sponge replica technique (SRT)

Ball clay slurry having distinctive clay content inside the range 35 to 55 wt.% with 0.4 wt.% sodium silicate as deflocculant has been prepared. Poly Vinyl Alcohol (PVA) 5wt.% as binder also used in different batch of slurry to keep the right viscosity of slurry. Cubic sponge of length 2.5×2.5×2.5 cm turned into immersed into the organized Ball clay slurry and the soaked samples of sponge had been dried at 80°C for 12 hours and then the dried samples have been fired at 1300°C and the fired samples were characterized by porosity, bloodless crushing electricity, and microstructure

Powder + water + deflocculant + with/ without PVA

(Ball clay) (Sod silicate)

↓

Pot Milling

↓

Stable Slurry

↓

Impregnation of Slurry into Sponge

↓

Drying

↓

Sintering

↓

Characterization

Fig. 2 Flowchart of sample preparation by Sponge Replica Technique

2.5 Characterizations of Porous Sample

2.5.1 Rheological Analysis

The rheological conduct of solid Ball clay slurries has been calculated by Anton Parr Rheometer. The experiments have been achieved with a growing shear rate (1-200 s-1) at 25°C. With the help of this Anton Parr Rheometer viscosity, shear pressure became calculated.

2.5.2 Porosity Calculation

The porosity of porous ceramic frame organized by using sponge reproduction technique has been calculated through vacuum method. In this technique first of all dry weight of samples were taken and then the samples positioned into a glass beaker full of kerosene. The beaker then put in a vacuum desiccator for approximately 2 hours interior vacuum after which the soaked weight and suspended weight were measured by stability. Apparent porosity and bulk density of samples have been calculated via the assist of given formulae

- *Apparent Porosit = Soaked Weight−Dry Weight/ Soaked Weight−Suspended Weight*
- *Bulk Density = Dry Weight ×Density of Kerosene / Soaked Weight−Suspended Weight*
- *Relative Density = Bulk Density× 100 / Theortical Density*

The theoretical density of Ball clay has been taken as 2.51gm/cc.

2.6 Measurement of Strength of Samples

The strength of the porous scaffold prepared by Sponge Replica Technique was measured. Force-deflection curve was plotted and the ultimate tensile strength was measured.

2.7 Microstructural Analysis

Microstructural analysis of porous samples has been done by a typical electron microscope i.e., Field Emission scanning Electron Microscope (FESEM) in which images of the surface of samples were taken by using electron beams. In this technique the significant information about the samples microstructure were obtained by the interaction of secondary electrons (SE), backscattered electron (BSE), as well as characteristic X-rays with the surface of porous body. The microstructure of porous samples was observed by using scanning electron microscope (Nova Nano SEM/FEI). The porous samples were coated to avoid charging.

3 RESULTS AND DISCUSSIONS

3.1 Raw Materials Characterization.

1) Particle Size Distribution of Clay.

The particle length distribution of ball clay powder as measured by Zeta sizer is given in Figure 3. From the figure, it is able to be observed that maximum particles are of the size in the range of 200-750nm. It can be visible that the graph is almost symmetric on each side of its maxima. The most particles length was located to be 400nm. The nature of the curve indicates a mono modal particle size distribution. Thus, the look at indicates that the ball clay used in the gift examine has a mono modal debris size distribution with a peak nearly about 400nm.

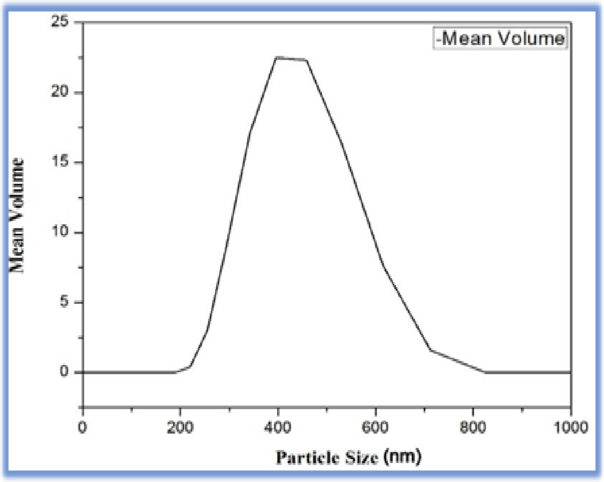

Figure. 1. Particle Size Distribution of clay

2) DSC-TG of Ball Clay

In the figure 4, the DSC –TG curve of ball clay powder has been seen. At about 100°C, there may be a top of the TG curve that is may be because of the disturbance within the machine at the time of beginning the experiment.

On heating ball clay physically, adsorbed water goes out at a temperature of approximately a 150°C. At about 450°C, the endothermic peak due to the dehydration of Ball clay (crystalline water goes out). At about 1050°C mullite formed and that is an exothermic reaction.

$Al2O3.\ 2\ SiO2.\ 2H2O \rightarrow Al2O3.\ 2\ SiO2 + 2H2O$

Due to the removal of crystalline water at around 450°C, there's a weight loss of approximately eight% and once more during mullite formation at around 1050°C, some weight loss located which is set 2-3%. The graph showing a few weights loss all through mullite formation that can't be defined because no weight loss takes place at some stage in mullite formation

$Al2O3.\ 2\ SiO2 \rightarrow 3Al2O3.\ 2\ SiO2$ (mullite)

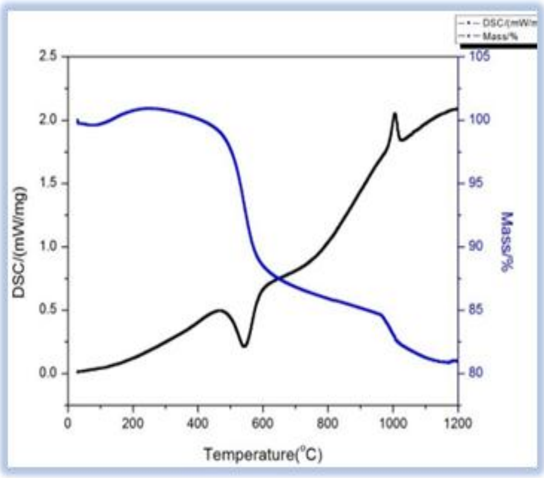

Figure 2. DSC-TG of ball clay

3) Effect of Temperature on Firing Shrinkage and Porosity of Ball Clay Sample

From the figure, it could be observed that from the temperature one thousand to 1200°C, porosity decreases gradually however from the temperature 1200 to 1350°C, the porosity decreases rapidly. This rapid change in porosity is due to the formation of liquid segment all through sintering. This liquid phase formed because of the presence of impurity in the substances. Thus, the samples of high solid loading have excessive packing density and coffee loading samples have low packing density for this reason excessive porosity.

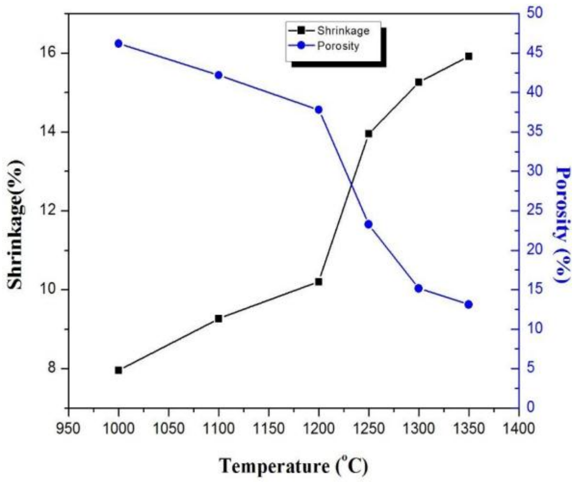

Figure.3. Effect of temperature on Porosity and Shrinkage

4) Optimization of slurry

- **Effect of Deflocculant on Zeta Potential**

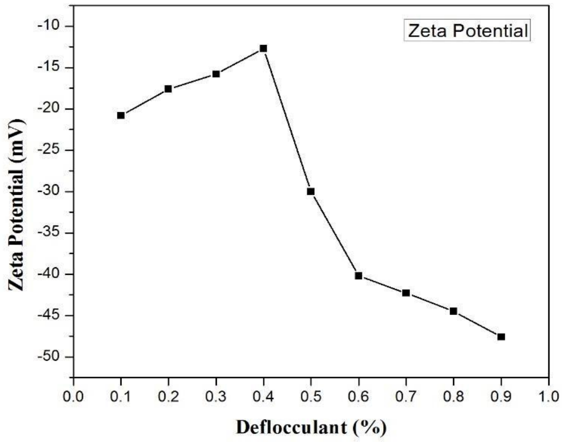

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Figure 4. Effect of deflocculant on Zeta Potential of Ball clay slurry

- **Effect of Deflocculant on Sedimentation height**

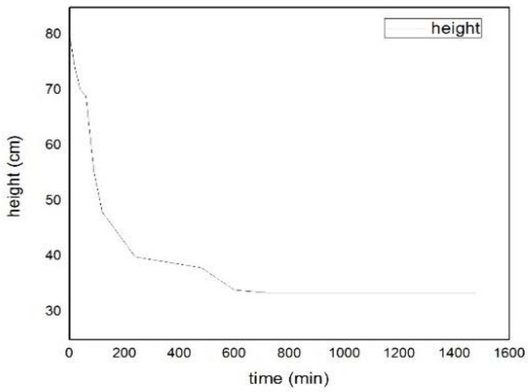


Figure 5. Rate of settling of particles

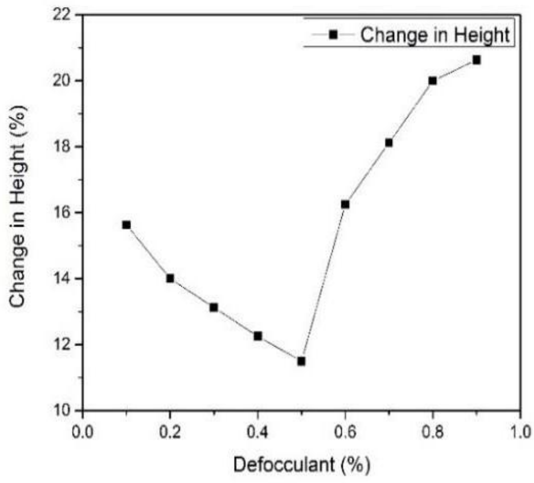
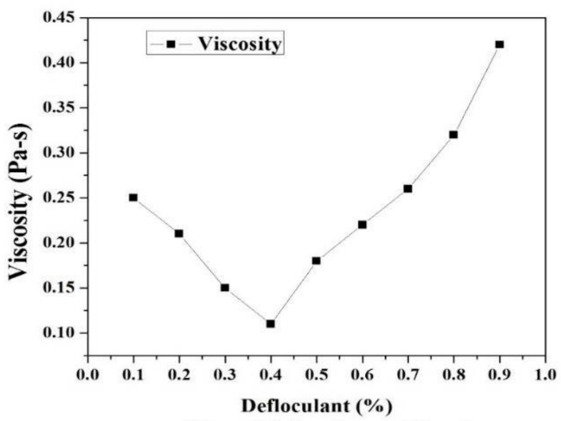


Figure 6. Effect of deflocculant on Sedimentation height

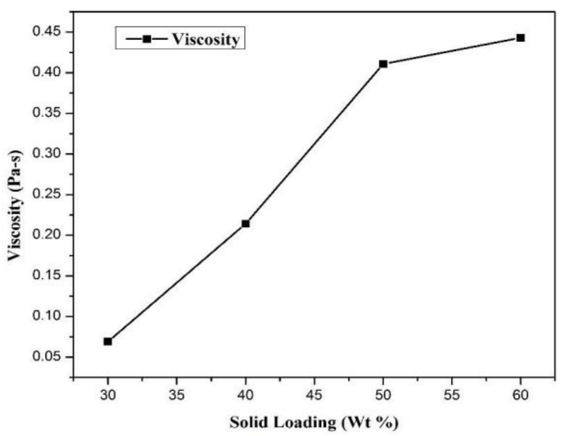
- **Effect of Deflocculant on Viscosity of slurry**



Effect of defloculant on Viscosity

- **Characterization of Porous Sample**

Effect of solid loading on viscosity



4 CONCLUSION

Ball clay utilized in present paintings have the most particles in the range of 250 - 800 nm. Hence the ball clay includes pleasant debris and pleasant in nature. From the XRD, it become discovered that the kaolinite is the fundamental phase found in ball clay and mullite is the essential segment inside the ball clay that is fired at 1300 for 2hrs. With the assist of Zeta Potential, Sedimentation top, and Viscosity, it turned into located that irrespective of stable loading of the slurry zero.4wt.% sodium silicate become sufficient to acquire a stable and dispersed slurry.

The porosity and electricity of the samples may be various from 60-85% and zero.02- zero.12MPa respectively with varying the solid loading from 40 – 60 wt.% on this method. No massive impact of PVA turned into observed on the houses of porous scaffolds.

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